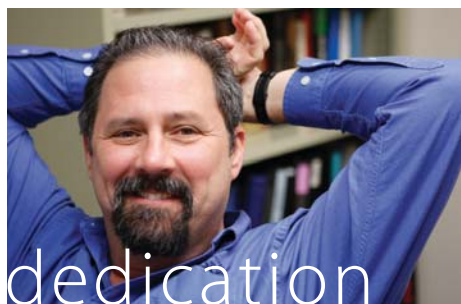


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Jack Cochran, Director of New Business and Technology



## Not All "624s" Are Equivalent

Improve Volatiles Analyses with  
New **Rxi®-624Sil MS** Columns:

- **Lower detection limits for active compounds**—See why Rxi®-624Sil MS columns improve sensitivity, accuracy, and MS performance.

- **Best-in-class G43**—Increase system suitability pass rates for USP <467> with the most selective G43 available.

- **Optimized method for volatile organics**—Minimize downtime by syncing instrument cycles with your purge and trap.

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Improve pass rates for USP <467> residual solvent analyses .....	4-5
Speed up volatiles analyses with synchronized GC conditions .....	6-7
Tools and accessories for your Rxi®-624Sil MS column .....	8-9
Single phase solutions for analyzing dietary supplements by LC .....	10-11
Food safety: 280 pesticide residues by LC/MS/MS .....	12-13
Take matrix out of the equation when analyzing diuretics .....	14-15
Increasing productivity for SimDist analyses .....	16-17
Unraveling scent signals to protect African wild dogs .....	18-19



## Not all "624s" are Equivalent Introducing Rxi®-624Sil MS Columns

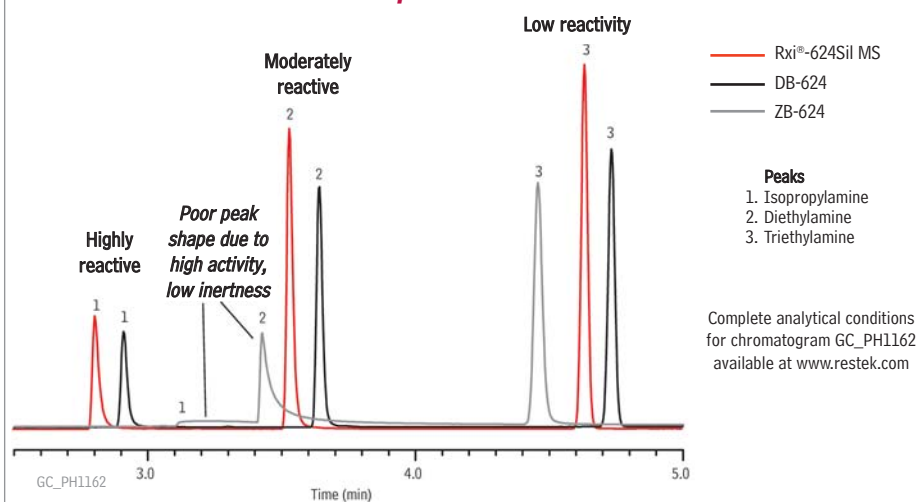
### New Rxi®-624Sil MS Columns Give Better Peak Shape, Improving Sensitivity

Whether you are developing methods for residual solvents, analyzing environmental VOCs, or running other applications for volatile organics, you can improve data quality with Rxi®-624Sil MS columns.

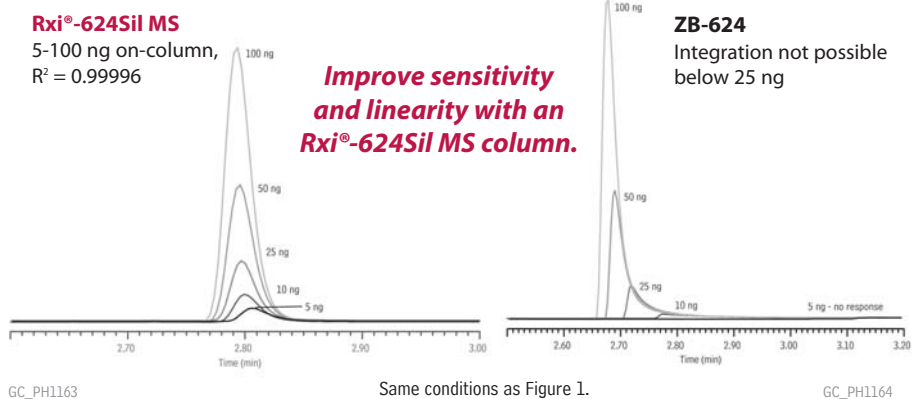
These new columns are more inert than other 624 type columns, resulting in higher response, better peak symmetry, and easier integration of active compounds (Figure 1). Since active analytes can be quantified at lower levels compared to similar products (Figure 2), Rxi®-624Sil MS columns are the best choice when increased sensitivity is desired.

**Figure 1** Highly inert Rxi®-624Sil MS columns provide better peak shape and simplify integration for active compounds at low levels (5 ng on-column).

**Rxi®-624Sil MS columns give more accurate results for active compounds.**



**Figure 2** Active compounds like isopropylamine can be more accurately integrated on an Rxi®-624Sil MS column, lowering levels of quantification (LOQs) and increasing accuracy.



get **more**

For more information on Rxi®624-Sil MS columns, download PHFL1245 at [www.restek.com](http://www.restek.com)

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### Lowest Bleed 624 Available—Assured GC/MS Compatibility

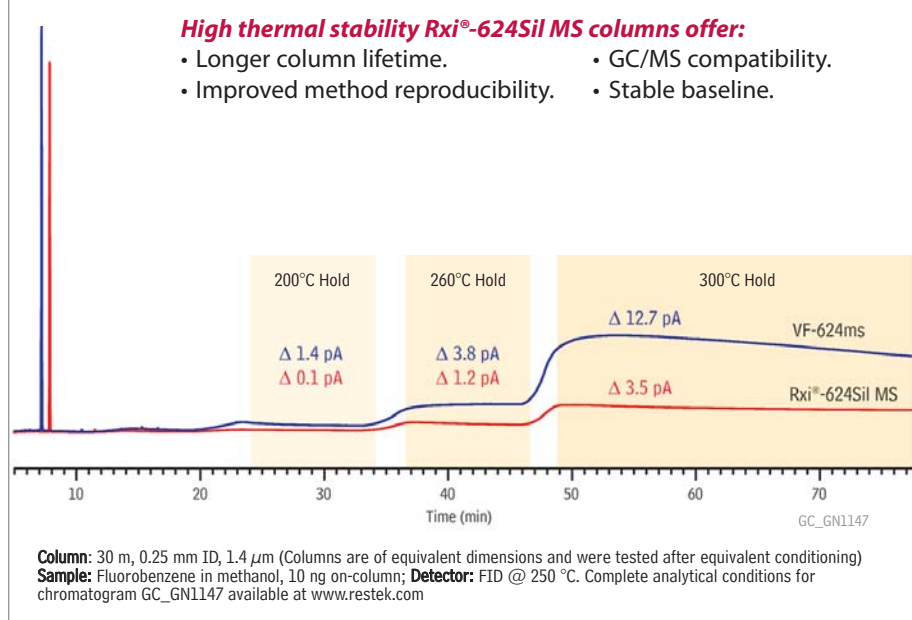
In addition to providing higher inertness and more accurate quantification of active compounds, Rxi®-624Sil MS columns offer greater thermal stability, resulting in lower bleed than any column in its class (Table I, Figure 3). While other 624 columns generate too much bleed to be useful for mass spec work, the Rxi®-624Sil MS column is fully compatible with mass spectrometry. Other benefits related to thermal stability include stable baselines, longer column lifetime, and improved method reproducibility.

**Table I** The Rxi®-624Sil MS column has the highest thermal stability of any 624 column.

Column	Manufacturer	Maximum Programmable Temperature
Rxi-624Sil MS	Restek	320 °C
VF-624ms	Varian	300 °C
DB-624	Agilent J&W	260 °C
ZB-624	Phenomenex	260 °C

Data obtained from company website or literature for a 30 m x 0.25 mm x 1.4 µm df column.

**Figure 3** The Rxi®-624Sil MS column has the lowest bleed of any column in its class and provides true GC/MS capability.



### Make your next Volatiles Column an Rxi®-624Sil MS Column

You can get more accurate low level results for volatile organics with a mass spec compatible Rxi®-624Sil MS column. See our articles in this issue for pharmaceutical (p. 4) and environmental (p. 6) applications, or contact us to discuss your own method needs.

### Rxi®-624Sil MS Columns (fused silica)

(mid polarity Crossbond® silarylene phase; equivalent to 6% cyanopropylphenyl/94% dimethyl polysiloxane)

ID	df (µm)	temp. limits	length	cat. #
0.18mm	1.00	-20 to 300/320°C	20-Meter	13865
0.25mm	1.40	-20 to 300/320°C	30-Meter	13868
0.32mm	1.80	-20 to 300/320°C	30-Meter	13870
0.32mm	1.80	-20 to 300/320°C	60-Meter	13872
0.53mm	3.00	-20 to 280/300°C	30-Meter	13871



Visit [www.restek.com/rxi](http://www.restek.com/rxi) for detailed comparisons and to learn how exceptional Rxi® inertness, bleed, and reproducibility can improve your data.



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3



## Improve Pass Rates for Residual Solvents by USP <467> With New Rxi®-624Sil MS GC Columns

By Rick Lake, Pharmaceutical Market Development Manager and Amanda Rigdon, Innovations Chemist

- Greatest resolution of acetonitrile and dichloromethane of any G43 column.
- Stable baseline for improved sensitivity of carbon tetrachloride.
- Exceptional column-to-column reproducibility.

It has been over a year since the revised USP <467> Residual Solvents general chapter became effective. Since then, many labs have experienced difficulty implementing this more expansive procedure. Most of the problems encountered relate to the selectivity and sensitivity needed to meet system suitability requirements in Procedure A. Finding an instrument setup that adequately resolves acetonitrile and dichloromethane in the Class 2 Mixture A standard, while also maintaining carbon tetrachloride sensitivity in the Class 1 solution, can prove difficult. Since increasing system suitability pass rates directly increases lab productivity, Restek has developed a new G43 capillary column that better meets USP <467> requirements.



get **more**

For more information on USP <467> analysis, download PHFL1018A at [www.restek.com](http://www.restek.com)

### Rxi®-624Sil MS Columns (fused silica)

(mid polarity Crossbond® silarylene phase; equivalent to 6% cyanopropylphenyl/94% dimethyl polysiloxane)

ID	df (µm)	temp. limits	length*	cat. #
0.18mm	1.00	-20 to 300/320°C	20	13865
0.25mm	1.40	-20 to 300/320°C	30	13868
0.32mm	1.80	-20 to 300/320°C	30	13870
0.32mm	1.80	-20 to 300/320°C	60	13872
0.53mm	3.00	-20 to 280/300°C	30	13871

\*Length in meters.

### Greater Resolution Improves Pass Rates

The Class 2 Mixture A solution contains the most difficult selectivity requirement of the method: the resolution between acetonitrile and dichloromethane must be greater than 1. This is often difficult to achieve on conventional G43 columns, which only give marginal selectivity for this pair. Poor selectivity can result in lower overall pass rates, and, thus, decreased sample throughput. In contrast, the Rxi®-624Sil MS column incorporates a distinctive bonding chemistry that results in resolution values consistently greater than 3 (Figure 1). The greater resolution routinely achieved on the Rxi®-624Sil MS column results in more consistent system suitability pass rates, and thus greater lab productivity.

### Higher Inertness Gives Increased Sensitivity

Rxi®-624Sil MS columns are manufactured using proprietary Rxi® technology, which produces extremely inert and stable columns. The high thermal stability of the Rxi®-624Sil MS column produces a very stable baseline, which leads to accurate and consistent integration. For example, carbon tetrachloride in the Class 1 system suitability solution, the most difficult sensitivity requirement in the method, can be easily and consistently integrated, reliably providing the necessary sensitivity. Another significant advantage of the Rxi®-624Sil MS column for the Class 1 solution is the complete resolution of benzene and 1,2-dichloroethane (Figure 2). Complete resolution of these analytes is often not achieved on other columns, making the Rxi®-624Sil MS column particularly beneficial for testing programs using USP <467>.

### Conclusion

Not all G43 columns are equivalent for residual solvent testing, and the new Rxi®-624Sil MS column offers best-in-class performance advantages for all aspects of USP <467> system suitability testing. These columns reliably produce improved resolution and sensitivity, increasing system suitability pass rates and ensuring more productive laboratory time.

For the complete version of this condensed article, visit [www.restek.com/adv001](http://www.restek.com/adv001)



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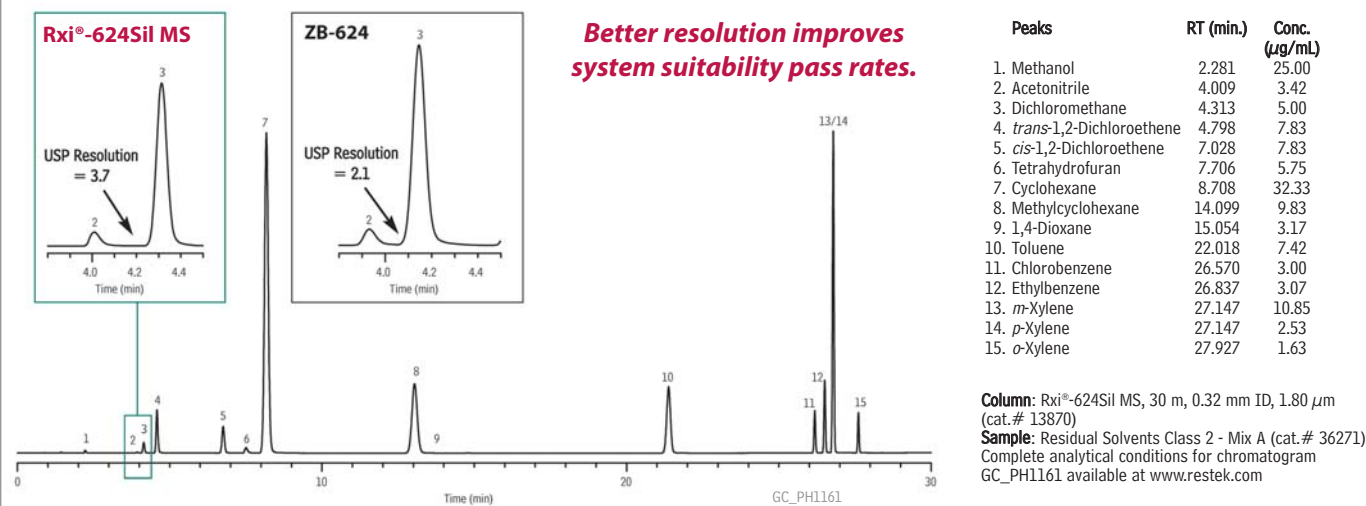
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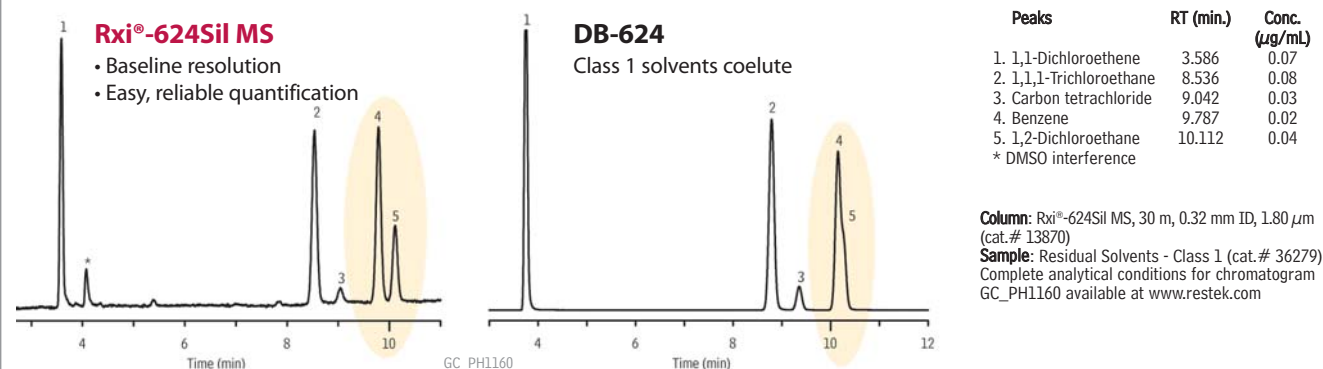
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**Figure 1** Rxi®-624Sil MS columns reliably resolve Class 2 Mix A compounds.



**Figure 2** The Rxi®-624Sil MS column provides complete resolution of the USP <467> Class 1 solution components—a result not often achieved on other G43 columns.



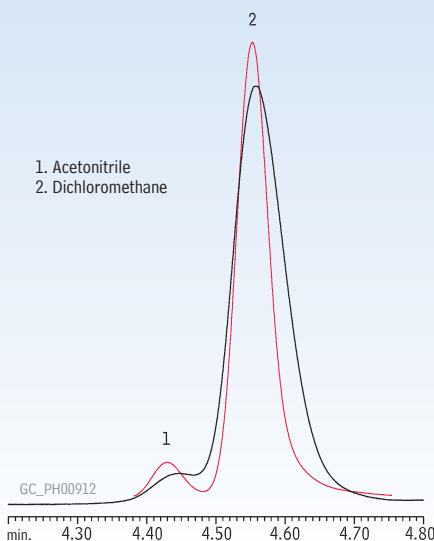
Tim Herring, Technical Service Specialist

## TECH TIP!

Resolution passes USP <467> criteria when using a 1mm liner (red line), but fails if a 4mm liner is used (black line).

When running USP <467> by headspace, using a **smaller bore liner (1 mm) can improve system suitability pass rates.** Larger bore liners (4 mm) are used with direct liquid injection because the sample is vaporized in the injection port and the liner must be able to accommodate the solvent expansion volume. In contrast, in headspace analysis, the sample is vaporized in a vial instead of the injection port, so a large volume liner is not needed, and in fact it can be deleterious. In headspace methods, using a smaller bore liner reduces band broadening by increasing linear velocity, allowing faster sample transfer and improving resolution.

See p. 9 for select 1mm liners.



Restek carries a full line of headspace essentials

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5





## Are Your Volatiles Methods Slowing You Down? Minimize Downtime with an Rxi®-624Sil MS and Our Synchronized GC Conditions

By Michelle Misselwitz, Innovations Chemist, Gary Stidsen, Product Manager, and Chris English, Innovations Manager

- Optimized analysis allows for 36 runs per 12-hour shift, increasing instrument productivity.
- Rxi®-624Sil MS column selectivity and inertness resolve critical pairs.
- High temperature stability reduces bleed profile, resulting in lower detection limits.

Maximizing sample throughput while maintaining adequate resolution can be a delicate balancing act when analyzing volatile organic compounds (VOCs). Conditions optimized for resolution can result in long analysis times, but using faster run times can result in problematic coelutions. Often, "624" type columns are chosen for their selectivity, but thermal stability is usually poor, resulting in phase bleed that decreases detector sensitivity. New Rxi®-624Sil MS columns offer reliable resolution of VOCs and also provide lower bleed and greater inertness than other 624 columns. Labs interested in optimizing sample throughput and

resolution can adopt the synchronized conditions established here on Rxi®-624Sil MS columns to maximize productivity and assure accurate, reliable results.

### *Want lower detection limits for active compounds?*

See page 2 to learn why Rxi®-624Sil MS columns improve sensitivity, accuracy, and MS performance for active analytes.

### **Reduce Downtime and Resolve Critical Pairs**

In order to minimize downtime between injections while ensuring good resolution, we established parameters that synchronized the purge and trap and instrument cycles while maintaining desired separations. Several critical pairs were chosen for computational modeling using Pro ezGC software. The initial temperature program determined by the software provided the best resolution, but resulted in an analysis time of 19 minutes. Since the purge and trap cycle time was 16.5 minutes, we tested other conditions to see if adequate resolution could be maintained using a faster instrument cycle. The program shown in Figure 1 reduced instrument downtime by better synchronizing injection and analysis, and also provided excellent resolution. Using a highly inert, low bleed Rxi®-624Sil MS column under the conditions established here, optimizes sample throughput while assuring good resolution of volatile organic compounds.

For the complete version of this condensed article, visit [www.restek.com/adv002](http://www.restek.com/adv002)

### **Rxi®-624Sil MS Columns (fused silica)**

(mid polarity Crossbond® silarylene phase; equivalent to 6% cyanopropylphenyl/94% dimethyl polysiloxane)

ID(mm)	df (μm)	temp. limits	length*	cat. #
0.18	1.00	-20 to 300/320°C	20	13865
0.25	1.40	-20 to 300/320°C	30	13868
0.32	1.80	-20 to 300/320°C	30	13870

\*Length in meters

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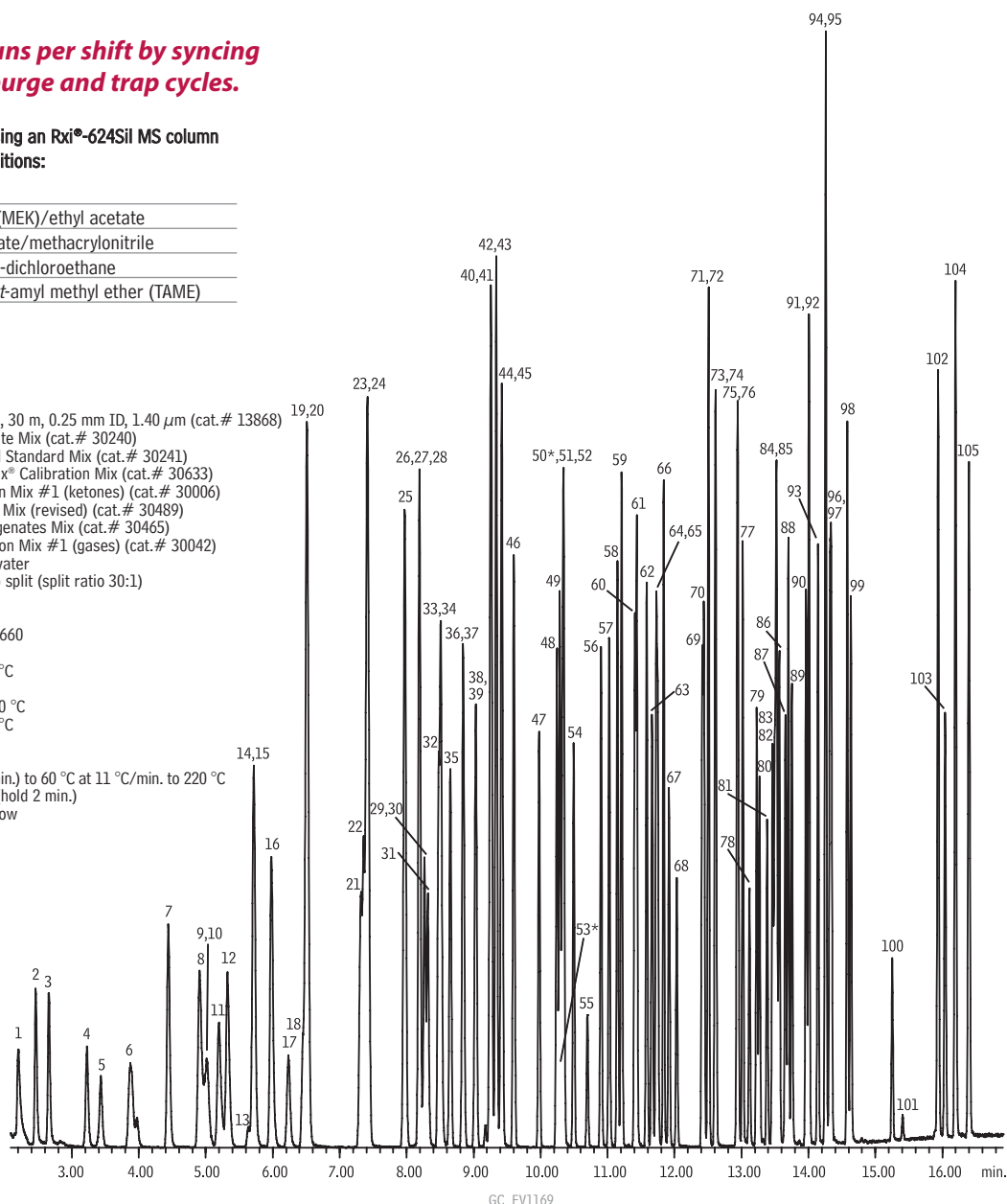
**Figure 1** Using an Rxi®-624Sil MS column under optimized conditions assures good resolution with minimal downtime.

**Analyze up to 36 runs per shift by syncing instrument and purge and trap cycles.**

Critical pairs resolved using an Rxi®-624Sil MS column under synchronized conditions:

Peak #s	Compounds
26/29	2-butanone (MEK)/ethyl acetate
31/32	methyl acrylate/methacrylonitrile
41/42	benzene/1,2-dichloroethane
41/45	benzene/ <i>tert</i> -amyl methyl ether (TAME)

**Column:** Rxi®-624Sil MS, 30 m, 0.25 mm ID, 1.40 µm (cat.# 13868)  
**Sample:** 8260A Surrogate Mix (cat.# 30240)  
 8260A Internal Standard Mix (cat.# 30241)  
 8260B MegaMix® Calibration Mix (cat.# 30633)  
 VOA Calibration Mix #1 (ketones) (cat.# 30006)  
 8260B Acetate Mix (revised) (cat.# 30489)  
 California Oxygenates Mix (cat.# 30465)  
 502.2 Calibration Mix #1 (gases) (cat.# 30042)  
**Conc.:** 25 ppb in RO water  
**Injection** purge and trap split (split ratio 30:1)  
**Inj. Temp.:** 225 °C  
**Purge and Trap**  
**Instrument:** OI Analytical 4660  
**Trap Type:** 10 Trap  
**Purge:** 11 min. @ 20 °C  
**Desorb Preheat Temp.:** 180 °C  
**Desorb:** 0.5 min. @ 190 °C  
**Bake:** 5 min. @ 210 °C  
**Interface Connection:** injection port  
**Oven**  
**Oven Temp:** 35 °C (hold 5 min.) to 60 °C at 11 °C/min. to 220 °C at 20 °C/min. (hold 2 min.)  
**Carrier Gas:** He, constant flow  
**Flow Rate:** 1.0 mL/min.  
**Detector:** MS  
**Mode:** Scan  
**Transfer Line Temp.:** 230 °C  
**Analyzer Type:** Quadrupole  
**Source Temp.:** 230 °C  
**Quad Temp.:** 150 °C  
**Electron Energy:** 70 eV  
**Solvent Delay Time:** 1.5 min.  
**Tune Type:** BFB  
**Ionization Mode:** EI  
**Scan Range:** 36-260 amu  
**Instrument:** Agilent 7890A GC & 5975C MSD  
**Notes**  
 Other Purge and Trap Conditions:  
 Sample Inlet: 40 °C  
 Sample: 40 °C  
 Water Management:  
 Purge 110 °C, Desorb 0 °C, Bake, 240 °C



Peaks	RT (min.)	20. <i>trans</i> -1,2-Dichloroethene	6.512	44. Isobutyl alcohol	9.421	66. Butyl acetate	11.837	90. <i>tert</i> -Butylbenzene	13.965
1. Dichlorodifluoromethane (CFC-12)	2.198	21. 1,1-Dichloroethane	7.315	45. <i>tert</i> -Amyl methyl ether (TAME)	9.421	67. Dibromochloromethane	11.921	91. Pentachloroethane	14.007
2. Chloromethane	2.459	22. Vinyl acetate	7.359	46. Fluorobenzene	9.598	68. 1,2-Dibromoethane (EDB)	12.035	92. 1,2,4-Trimethylbenzene	14.010
3. Vinyl chloride	2.659	23. Diisopropyl ether (DIPE)	7.407	47. Trichloroethene	9.976	69. Chlorobenzene-d5	12.412	93. <i>sec</i> -Butylbenzene	14.140
4. Bromomethane	3.226	24. Chloroprene	7.429	48. 1,2-Dichloropropane	10.243	70. Chlorobenzene	12.440	94. 4-Isopropyltoluene ( <i>p</i> -cymene)	14.254
5. Chloroethane	3.434	25. Ethyl <i>tert</i> -butyl ether (ETBE)	7.970	49. Methyl methacrylate	10.290	71. Ethylbenzene	12.507	95. 1,3-Dichlorobenzene	14.263
6. Trichlorofluoromethane (CFC-11)	3.876	26. 2-Butanone (MEK)	8.193	50. 1,4-Dioxane (ND)	10.299*	72. 1,1,1,2-Tetrachloroethane	12.507	96. 1,4-Dichlorobenzene-D4	14.321
7. Diethyl ether (ethyl ether)	4.440	27. <i>cis</i> -1,2-Dichloroethene	8.193	51. Dibromomethane	10.326	73. <i>m</i> -Xylene	12.612	97. 1,4-Dichlorobenzene	14.340
8. 1,1-Dichloroethene	4.909	28. 2,2-Dichloropropane	8.265	52. Propyl acetate	10.346	74. <i>p</i> -Xylene	12.935	98. <i>n</i> -Butylbenzene	14.579
9. 1,1,2-Trichlorotrifluoroethane (CFC-113)	4.998	29. Ethyl acetate	8.276	53. 2-Chloroethanol (ND)	10.368*	75. <i>o</i> -Xylene	12.949	99. 1,2-Dichlorobenzene	14.635
10. Acetone	5.029	30. Propionitrile	8.318	54. Bromodichloromethane	10.496	76. Styrene	13.018	100. 1,2-Dibromo-3-chloropropane (DBCP)	15.252
11. Iodomethane	5.195	31. Methyl acrylate	8.476	55. 2-Nitropropane	10.698	77. <i>n</i> -Amyl acetate	13.118	101. Nitrobenzene	15.407
12. Carbon disulfide	5.323	32. Methacrylonitrile	8.507	56. <i>cis</i> -1,3-Dichloropropene	10.904	78. Bromoform	13.226	102. 1,2,4-Trichlorobenzene	15.935
13. Acetonitrile	5.637	33. Bromochloromethane	8.521	57. 4-Methyl-2-pentanone (MIBK)	11.026	79. Isopropylbenzene (cumene)	13.268	103. Hexachloro-1,3-butadiene	16.040
14. Allyl chloride	5.715	34. Tetrahydrofuran	8.543	58. Toluene-D8	11.148	80. <i>cis</i> -1,4-Dichloro-2-butene	13.385	104. Naphthalene	16.196
15. Methyl acetate	5.723	35. Chloroform	8.843	59. Toluene	11.210	81. 4-Bromofluorobenzene	13.456	105. 1,2,3-Trichlorobenzene	16.396
16. Methylene chloride	5.981	36. 1,1,1-Trichloroethane	8.848	60. <i>trans</i> -1,3-Dichloropropene	11.407	82. 1,1,2,2-Tetrachloroethane	13.515		
17. <i>tert</i> -Butyl alcohol	6.234	37. Dibromofluoromethane	9.026	61. Ethyl methacrylate	11.435	83. <i>trans</i> -1,4-Dichloro-2-butene	13.526		
18. Acrylonitrile	6.451	38. Carbon tetrachloride	9.037	62. 1,1,2-Trichloroethane	11.585	84. Bromobenzene	13.657		
19. Methyl <i>tert</i> -butyl ether (MTBE)	6.509	39. 1,1-Dichloropropene	9.246	63. Tetrachloroethene	11.662	85. 1,2,3-Trichloropropane	13.699		
		40. 1,2-Dichloroethane-d4	9.262	64. 1,3-Dichloropropane	11.729	86. <i>n</i> -Propylbenzene	13.751		
		41. Benzene	9.334	65. 2-Hexanone	11.749	87. 2-Chlorotoluene			
		42. 1,2-Dichloroethane	9.340			88. 1,3,5-Trimethylbenzene			
		43. Isopropyl acetate				89. 4-Chlorotoluene			

\* ND = not detected; retention time determined by wet needle injection

# Perfect Complements

## To Your New Rxi®-624Sil MS GC Column

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For other instrument specific EZ No-Vent® connectors, visit [www.restek.com/eznovent](http://www.restek.com/eznovent)

Description	qty.	cat.#
EZ No-Vent Connector Kit includes: EZ No-Vent Connector, two 0.4mm ID adaptor ferrules for capillary column, two 0.4mm ID ferrules for transfer line, 100µm deactivated transfer line (3 ft.), column plug, column nut	kit	21323
Replacement ferrules for connecting capillary column to EZ No-Vent Connector: 0.4mm ID (Polyimide)	2-pk.	21015
0.5mm ID (Polyimide)	2-pk.	21016
Replacement ferrules for connecting transfer line to EZ No-Vent Connector: 0.4mm ID	2-pk.	21043
Replacement 100µm deactivated transfer line	3 ft.	21018
Replacement EZ No-Vent Column Nut	5-pk.	21900
Replacement EZ No-Vent Plug	2-pk.	21915
Open-End Wrenches, 1/4" x 5/16"	2-pk.	20110

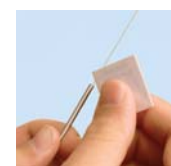
### Capillary Installation Gauge for Agilent 5973/5975 MS



**Easily seat ferrules for consistent installations in Agilent 5973 MS.**



Install the nut and ferrule onto the column, then insert the column through the installation tool, exposing several centimeters at the exit end, then tighten the nut on to the gauge.



Score and remove the exposed end of the column, then loosen the nut.



Ready to go!

Description	qty.	cat.#
Capillary Installation Gauge for Agilent 5973/5975 MS	ea.	21894

**Capillary gauges for other instrument manufacturers are available. Visit us online.**

### GC/MS Cleaning Kit



The Restek GC/MS Cleaning Kit (cat.#s 27194, 27195) includes:

- lint free nylon gloves (small-2pair)
- lint free nylon gloves (large-2 pair)
- lint free cotton cloth, 9 x 9 (10-pk.)
- micro mesh 4 x 6 sheet (4-pk.)
- aluminum oxide (75 gram jar)
- cotton tip applicators
- tweezers, large
- tweezers, small
- septum puller
- Dremel® tool, battery-operated (optional, 27194)
- tool kit bag

Poor sensitivity, loss of sensitivity at high masses, or high multiplier gain during an auto tune are all indicators that your mass spectrometer source may need to be cleaned. Restek has assembled all of the necessary components for cleaning and polishing your ion source.

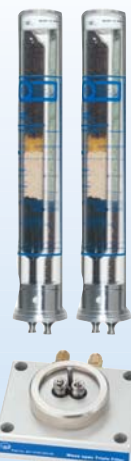
Description	qty.	cat.#
Mass Spec Cleaning Kit with Dremel Tool	kit	27194
Mass Spec Cleaning Kit without Dremel Tool	kit	27195
Mass Spec Cleaning Kit Replacement Parts Kit (includes cloths, micro mesh sheets, small and large gloves)	kit	27196

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Description	qty.	cat.#
<b>Electron Multipliers for Agilent GC/MS and LC/MS</b>		
For Agilent 5970 GC/MS	ea.	23072
For Agilent 5971, 5972, GC GC/MS	ea.	23073
For Agilent 5973 & 5975 GC/MS (includes mount for initial installation)*†	ea.	23074
For Agilent 5973 & 5975 GC/MS and LC/MSD (Replacement Multiplier)*†	ea.	23075
For Agilent LC/MSD (includes mount for initial installation)*†	ea.	23076
<b>Electron Multiplier for Applied Biosystems (Sciex)</b>		
For API 300, 3000 & 4000 Applied Biosystems	ea.	23077
<b>Electron Multiplier for Thermo Finnigan GC/MS</b>		
For Thermo TRACE DSQ, DSQII, and Polaris-Q GC/MS	ea.	23081

\*First time installation requires a mount which includes the mechanical housing. After initial installation, only the replacement electron multiplier is required.

†This unit is designed for use in the 5975, 5973 GC and the LC/MSD.

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## Split Liners for Agilent GCs

ID* x OD & Length	qty.	cat.#
1mm Split**		
1.0mm x 6.3mm x 78.5mm	ea.	20972
1.0mm x 6.3mm x 78.5mm	5-pk.	20973

## Zero Dilution Liners for PerkinElmer Auto SYS™ and Clarus GCs

ID* x OD & Length	qty.	cat.#
Zero Dilution Inner Liner		
1.0mm x 2.0mm x 73mm	ea.	22990
1.0mm x 2.0mm x 73mm	5-pk.	22991

## Split Liners for Shimadzu GCs

ID* x OD & Length	qty.	cat.#
1mm Split		
1.0mm x 5.0mm x 95mm	ea.	20976
1.0mm x 5.0mm x 95mm	5-pk.	20977
1.0mm x 5.0mm x 95mm	25-pk.	20978

## Split Liners for Varian 1075/1077 GCs

ID* x OD & Length	qty.	cat.#
1mm Split		
1.0mm x 6.3mm x 72mm	ea.	20970
1.0mm x 6.3mm x 72mm	5-pk.	20971

\*Nominal ID at syringe needle expulsion point.

\*\*Use this liner for increased sensitivity.

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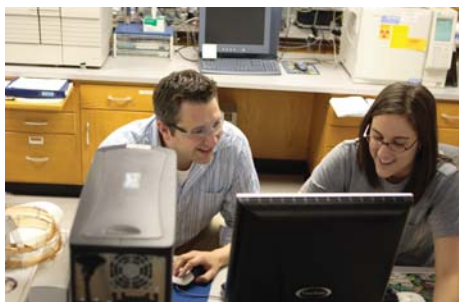
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dietary supplements



## Aqueous C18 LC Columns—More Versatile than a C18 for Vitamins and Organic Acids in Dietary Supplements

Ty W. Kahler, Innovations Chemist and Rick Lake, Pharmaceutical Market Development Manager

- Simplify method development for polar compounds.
- Higher retention and selectivity compared to a C18.
- Compatible with 100% aqueous mobile phases.

Conventional alkyl (C18) columns are frequently used for initial method development, but often are not the best choice. C18 columns have poor retention for polar compounds and do not perform well with aqueous mobile phases. In contrast, Aqueous C18 columns are a more versatile choice, due to much higher polar retention and compatibility with 100%

aqueous mobile phases. In this article, we demonstrate the utility of Aqueous C18 columns across a range of analytes relevant to dietary supplement testing.

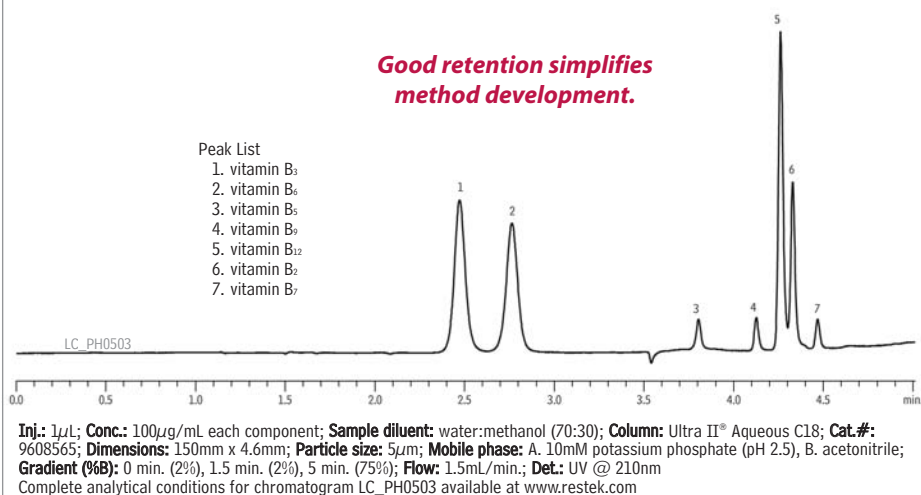
### Ideal for Multi-Vitamin Analyses—Easily Retains Water-Soluble Vitamins

Many consumers are augmenting their diets with multi-vitamins. These supplements usually contain multiple water-soluble vitamins in a variety of chemical forms and concentrations. While water-soluble vitamins can be analyzed by HPLC, obtaining adequate retention of hydrophilic analytes is often problematic. As shown in Figure 1, the Ultra II® Aqueous C18 column provides excellent retention and completely resolves a test mix of B vitamins.

## Need to test for pesticides too?

See p.12 for an LC/MS/MS analysis of 280 pesticides on an Ultra Aqueous C18 column.

**Figure 1** A selective separation of seven B vitamins using a simple buffer:acetonitrile gradient on an Ultra II® Aqueous C18 column.



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## Better for Organic Acids—More Retentive, Selective and Stable than a C18

Aqueous C18 columns are also an excellent choice for analyzing organic acids. For example, in Figure 2, an Ultra II® Aqueous C18 column provides greater retention and selectivity for organic acids than a conventional C18. The unique bonding chemistry of an Aqueous C18 column improves the retention of polar compounds and allows 100% aqueous mobile phases to be used, making it an excellent choice when developing methods for dietary supplement testing.

For the complete version of this condensed article, visit [www.restek.com/adv003](http://www.restek.com/adv003)

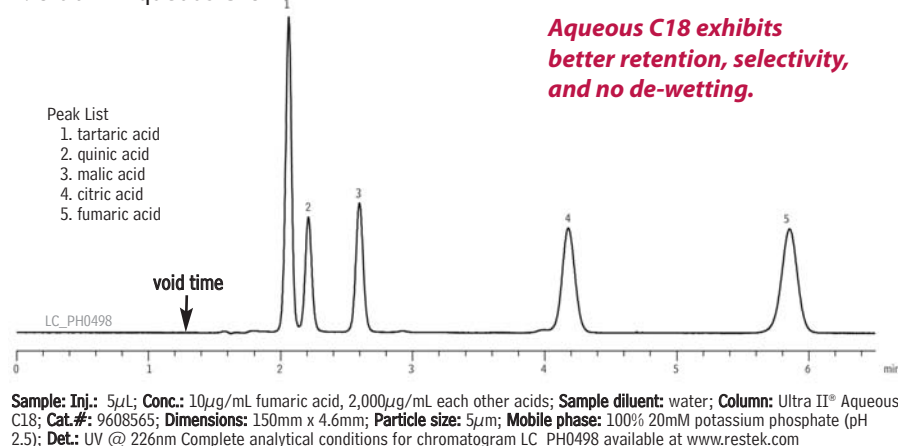
## Learning LINKS

Why use a reversed phase column specifically designed for highly aqueous mobile phases?

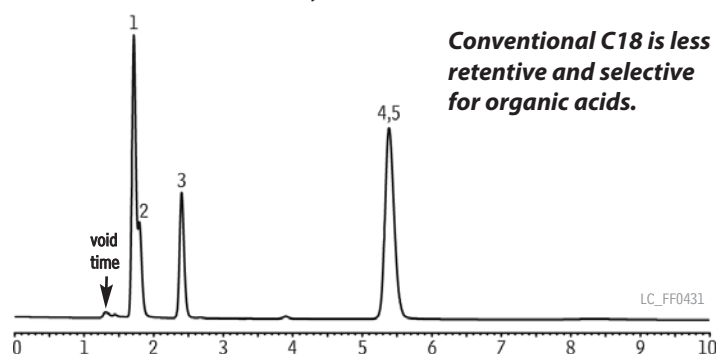
Learn why at [www.restek.com/adv008](http://www.restek.com/adv008)

**Figure 2** Ultra II® Aqueous C18 columns outperform conventional C18 columns for the analysis of organic acids in a 100% aqueous mobile phase.

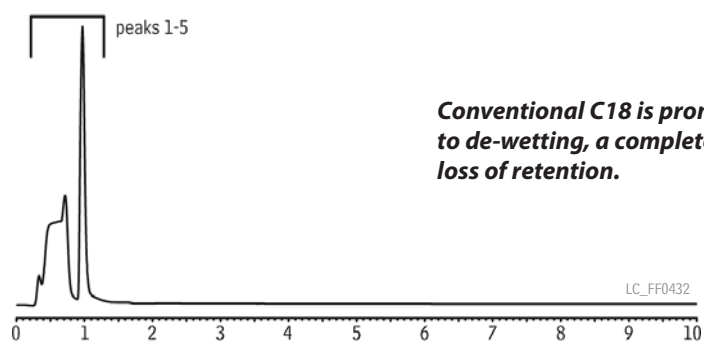
### A: Ultra II® Aqueous C18



### B: Conventional C18, initial analysis



### C: Conventional C18, post pressure release



Conditions used in A, B, and C were identical. De-wetting in Figure 1C was caused for illustrative purposes by releasing column pressure.

## Ultra II® Aqueous C18 Columns (USP L1)



### Physical Characteristics:

particle size: 2.2µm, 3µm or 5µm, spherical  
pore size: 100Å  
carbon load: 15%  
endcap: no  
pH range: 2.5 to 7.5  
temperature limit: 80°C

### Chromatographic Properties:

Highly retentive and selective for reversed phase separations of polar analytes. Highly base-deactivated. Compatible with highly aqueous (up to 100%) mobile phases.

	3.2mm ID	4.6mm ID
Length	cat.#	cat.#
<b>3µm Columns</b>		
30mm	9608333	9608335
50mm	9608353	9608355
100mm	9608313	9608315
150mm	9608363	9608365
<b>5µm Columns</b>		
30mm	9608533	9608535
50mm	9608553	9608555
100mm	9608513	9608515
150mm	9608563	9608565
200mm	9608523	9608525
250mm	9608573	9608575

More dimensions available online.

## Fruit Juice Organic Acid Standard

(5 components)

citric acid	2,000µg/ml	quinic acid	2,000
fumaric acid	10*	tartaric acid	2,000
malic acid	2,000		

In water, 1mL/ampul

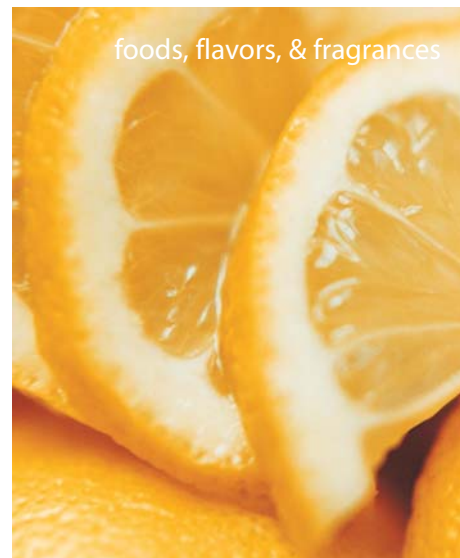
cat. # 35080 (ea.)

In water, 5mL/ampul

cat. # 35081 (ea.)

\*Fumaric acid is a trace impurity in malic acid, as well as an added component of the mix. The amount of fumaric acid in malic acid will not affect the stated concentration of malic acid, but can represent a significant and variable deviation from the low concentration of fumaric acid stated to be in the mix. All other components of the mix are at the specified concentration. Quantity discounts not available.





## Comprehensive Pesticide Residue Analysis by LC/MS/MS Using an Ultra Aqueous C18 Column

By Becky Wittrig, Ph.D., AB Sciex, and André Schreiber, Ph.D., Applied Biosystems/MDS Analytical Technologies

- Easily resolve and quantify more than 280 pesticide species.
- Use LC/MS/MS to reliably monitor difficult polar and/or thermally unstable species.
- Aqueous C18 phase offers optimal selectivity and retention.

Food safety is a topic of great interest globally. With recent contamination issues in a wide range of commodities, ensuring the quality of our food supply is becoming increasingly important. Pesticide residue content is one area of concern.

While pesticides have typically been monitored by gas chromatography, polar and/or thermally unstable pesticides are difficult or impossible to monitor using this approach. Thus,

traditional HPLC techniques are used for select pesticide classes, such as the carbamate and phenylurea pesticides.

### Ultra Aqueous C18 Columns (USP L1)



#### Physical Characteristics:

particle size: 3 $\mu$ m or 5 $\mu$ m, spherical  
pore size: 100Å  
carbon load: 15%  
endcap: no  
pH range: 2.5 to 7.5  
temperature limit: 80°C

#### Chromatographic Properties:

Highly retentive and selective for reversed phase separations of polar analytes. Highly base-deactivated. Compatible with highly aqueous (up to 100%) mobile phases.

Length	1.0mm ID cat. #	2.1mm ID cat. #
<b>3<math>\mu</math>m Columns</b>		
30mm	9178331	9178332
50mm	9178351	9178352
100mm	9178311	9178312
<b>5<math>\mu</math>m Columns</b>		
30mm	9178531	9178532
50mm	9178551	9178552
100mm	9178511	9178512
150mm	9178561	9178562
200mm	9178521	9178522
250mm	9178571	9178572

More dimensions available online.

With recent advances in LC/MS/MS instrumentation, this technique is quickly gaining acceptance for pesticide residue testing. LC/MS/MS can be used to simultaneously monitor hundreds of potential contaminants—including those difficult to detect by GC. Using both LC/MS/MS and GC approaches allows for a faster, more complete picture of pesticide residues. MS/MS technology also permits identification of the target pesticides through the selection of specific MRM transitions for each compound. For example, aldicarb, a carbamate pesticide, uses two MRM transitions of 208.2→89.1amu and 208.2→116.1amu.

While the MS/MS detector allows for specific, sensitive detection of the pesticide species, the LC separation is still important to ensure the highest quality data. Conventional C18 stationary phases are typically used for pesticide monitoring, but the selectivity and retention is poor for more polar species. In contrast, Ultra Aqueous C18 columns are ideal for multi-pesticide residue monitoring methods. In Figure 1, the analysis of more than 280 pesticides using the 3 $\mu$ m Ultra Aqueous C18 is shown. Optimized stationary phase selectivity allows for an even distribution of the compounds throughout the retention time window (see [www.restek.com/adv004](http://www.restek.com/adv004) for peak lists and retention times). As well, retention of more polar pesticides is greatly improved, as demonstrated in Figure 1C. The Ultra Aqueous C18 column, in a 100 x 2.1mm, 3 $\mu$ m configuration is the column of choice for LC/MS/MS pesticide monitoring methods.

Using LC/MS/MS technology and Aqueous C18 columns, in combination with gas chromatography, results in the most comprehensive monitoring of pesticide residues. Labs interested in more complete multi-residue analysis of pesticides in food matrices, including difficult polar or thermally unstable compounds, should consider adding LC/MS/MS and Aqueous C18 columns to routine testing procedures.

#### Acknowledgements

The authors wish to thank the US FDA for their collaboration and recognize the participation of multiple FDA labs in this work.

For the complete version of this condensed article, visit [www.restek.com/adv004](http://www.restek.com/adv004)

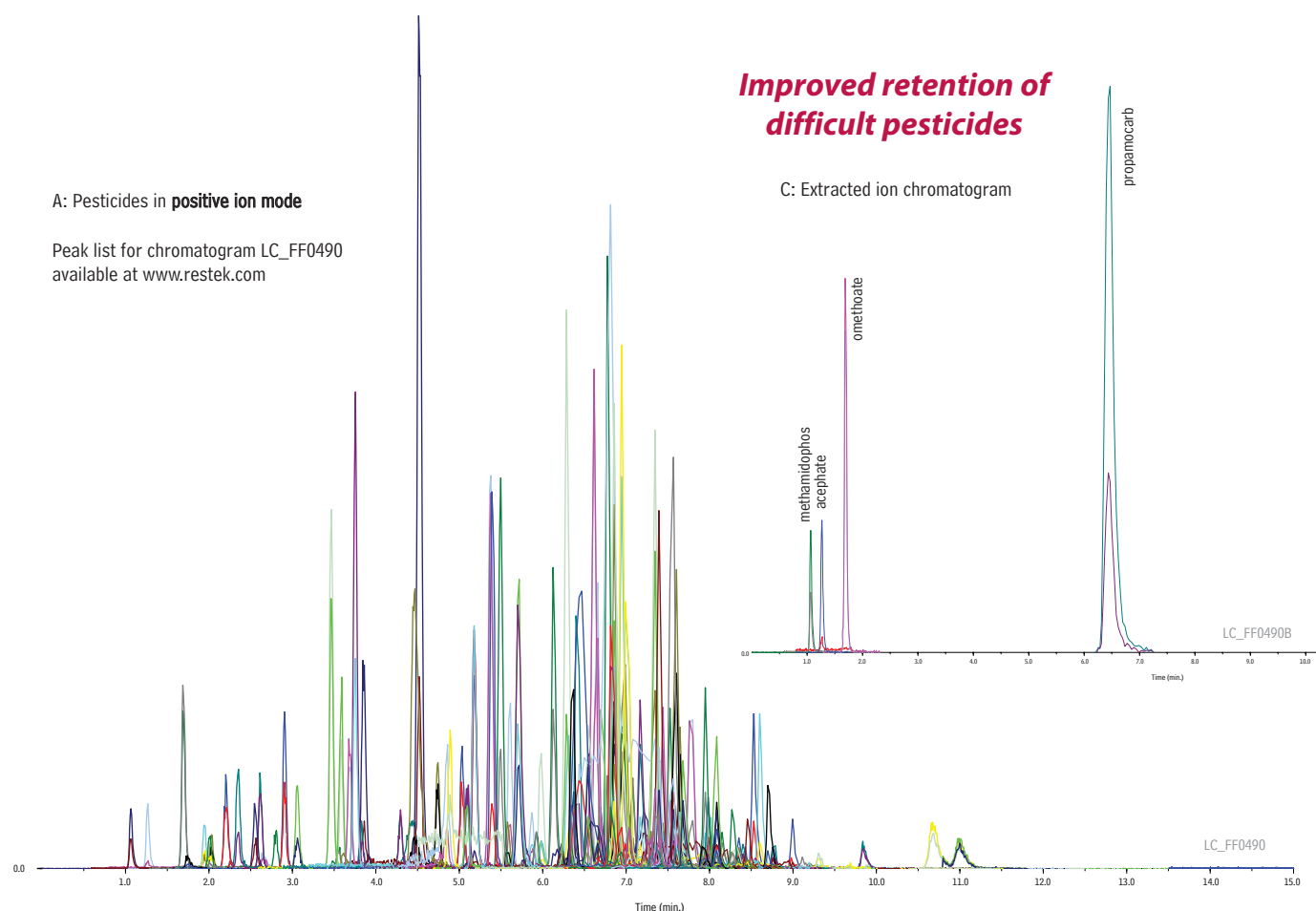
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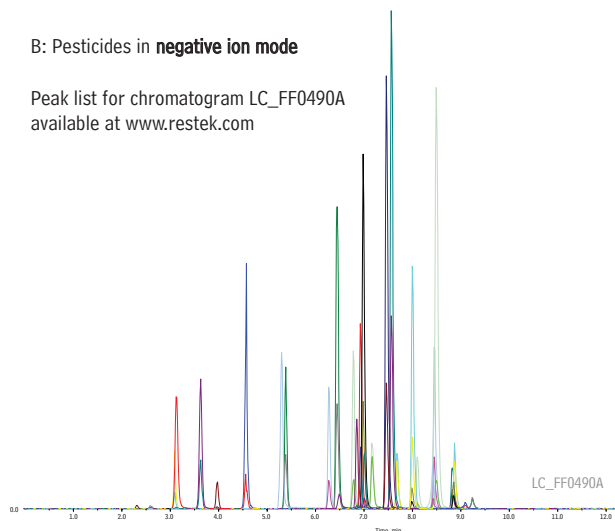
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**Figure 1** Easily monitor over 280 pesticides by LC/MS/MS.



**B: Pesticides in negative ion mode**

Peak list for chromatogram LC\_FF0490A available at [www.restek.com](http://www.restek.com)



**Sample:** multicomponent pesticide standard; **Inj.:** 10µL; **Conc.:** 1ppb each pesticide; **Sample diluent:** acetonitrile; **Column:** Ultra Aqueous C18; **Cat. #:** 9178312; **Dimensions:** 100mm x 2.1mm; **Particle size:** 3µm; **Pore size:** 100Å; **Instrument:** Shimadzu Prominence® UFLCxx; **Mobile phase:** 10 mM NH<sub>4</sub>OAc in water, B: 10 mM NH<sub>4</sub>OAc in methanol; **Gradient (%B):** 0 min. (20%), 8.0 min. (90%), 12.0 min. (100%), 14.8 min. (100%), 14.9 min. (20%); **Flow:** 500µL/min; **Temp.:** 35°C; **Detector:** Applied Biosystems 4000 QTRAP® LC/MS/MS system; **Ion Source:** TurboIonSpray®, A & C: ESI+, B: ESI-; **IonSpray Voltage:** 5kV (ESI+), -4.2kV (ESI-); **Gas 1:** 50psi; **Gas 2:** 60psi; **Source Temp.:** 600°C.

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## LC/MS/MS Analysis of Diuretics in Urine: Biphenyl Column Takes Matrix Out of the Equation

By Amanda Rigdon, Clinical/Forensic Innovations Chemist, Takeo Sakuma, AB Sciex, and Becky Wittrig, Ph.D., AB Sciex

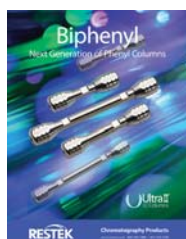
- Ultra II® Biphenyl columns separate compounds that coelute on phenyl hexyl columns.
- Improve quantitation through resolution of diuretics from isobaric matrix interferences.
- Fast analysis time supports high sample throughput.

Diuretics can mask the presence of performance enhancing drugs since they act to dilute the urine. Because of this, the use of diuretics has been banned by the World Anti-Doping Agency (WADA) and diuretic compounds are included in drug testing of athletes. Most common diuretics are highly functionalized compounds, making them hydrophilic and difficult to retain using C18 columns. Phenyl columns are a good alternative, as they generally have better hydrophilic retention; however, not all phenyl columns are retentive enough to ensure adequate resolution. While chromatographic resolution

is not always required for LC/MS/MS analyses, it is necessary when isobaric interferences are present, such as when testing for diuretics in urine.

### Better Retention Reduces Matrix Interference

Ultra II® Biphenyl columns can retain hydrophilic compounds longer than other phenyl-based stationary phases, due to the unique selectivity of the Biphenyl ligand for highly-functionalized aromatic compounds. As shown in Figure 2, using an Ultra II® Biphenyl column ensures complete separation of the diuretic amiloride from matrix peaks ( $k' = 5$ ). In contrast, matrix interference occurs on a Gemini® C6-Phenyl (phenyl hexyl) column ( $k' = 0.6$ ), preventing accurate quantitation. During this experiment, 10 diuretics from 4 classes were analyzed and excellent retention and resolution were obtained for all compounds in just 8 minutes, including re-equilibration time, on an Ultra II® Biphenyl column (see full chromatogram and conditions at [www.restek.com/adv005](http://www.restek.com/adv005)).



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For more information on biphenyl columns, download GNFL1277 at [www.restek.com](http://www.restek.com)

Despite the power of modern LC/MS/MS instrumentation, isobaric matrix interferences often complicate analyses involving biological samples and using a column that produces adequate retention is critical for accurate quantitation. An Ultra II® Biphenyl column, in combination with LC/MS/MS, provides fast, reliable results when analyzing diuretics in urine.

For the complete version of this condensed article, visit [www.restek.com/adv005](http://www.restek.com/adv005)



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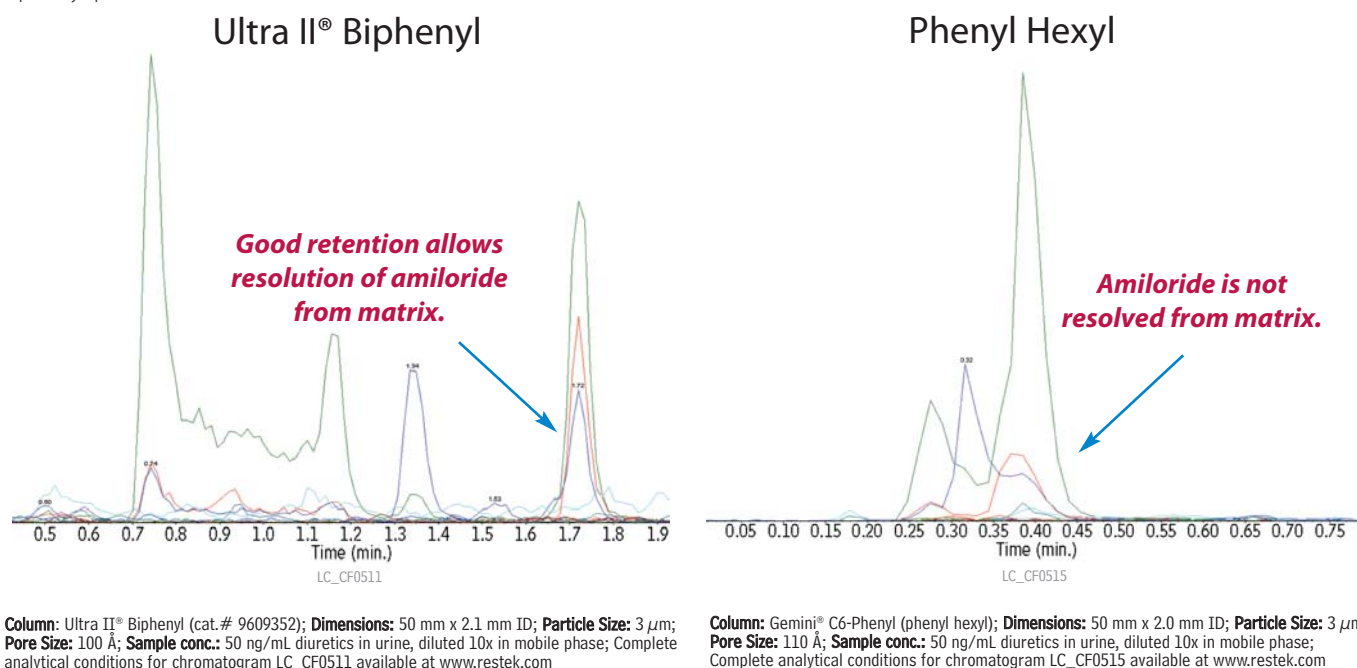
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**Figure 1** Higher retention on Ultra II® Biphenyl columns allows quantitation of diuretics that coelute with matrix on other phenyl phases.



## Ultra II® Biphenyl Columns (USP L11)



### Physical Characteristics:

**particle size:** 2.2µm, 3µm or 5µm, spherical  
**pore size:** 100Å  
**carbon load:** 15%  
**endcap:** fully endcapped  
**pH range:** 2.5 to 7.5  
**temperature limit:** 80°C

Length	1.0mm ID		2.1mm ID	
	cat.#		cat.#	
<b>1.9µm Columns</b>				
30mm	—	—	9609232	
50mm	—	—	9609252	
100mm	—	—	9609212	
<b>2.2µm Columns</b>				
30mm	—	—	9609832	
50mm	—	—	9609852	
100mm	—	—	9609812	
<b>3µm Columns</b>				
30mm	9609331		9609332	
50mm	9609351		9609352	
100mm	9609311		9609312	
150mm	9609361		9609362	
<b>5µm Columns</b>				
30mm	9609531		9609532	
50mm	9609551		9609552	
100mm	9609511		9609512	
150mm	9609561		9609562	
200mm	9609521		9609522	
250mm	9609571		9609572	

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## Increase Productivity: Get More Runs from Your SimDist Setup Using Next Generation MXT®-1HT Columns

By Jan Pijpelink, Petrochemical Market Development Manager and Barry Burger, Petrochemical Innovations Chemist

- Stable up to 450°C—lowest bleed for longest column lifetime.
- Reliably meet all ASTM D6352 and D7500 specifications.
- 100% dimethyl polysiloxane phase allows easy comparisons to historical data.

Accurate boiling point determination for medium and heavy fractions using GC simulated distillation requires columns and phase polymers that are robust enough to withstand high temperatures without significant degradation. Metal columns are a better alternative than fused silica, and the new MXT®-1HT SimDist columns are the lowest bleed, highest efficiency column available.

When compared to columns from other manufacturers, MXT®-1HT SimDist columns meet all D6352 method criteria and easily outperform competitors (Figure 1). In addition, field testing under accelerated conditions further demonstrates column robustness, even at 430°C (Figure 2). The exceptionally low bleed and high efficiency characteristics of the new MXT®-1HT SimDist columns translate directly into assured method performance, more analyses per calibration, and longer column lifetimes.

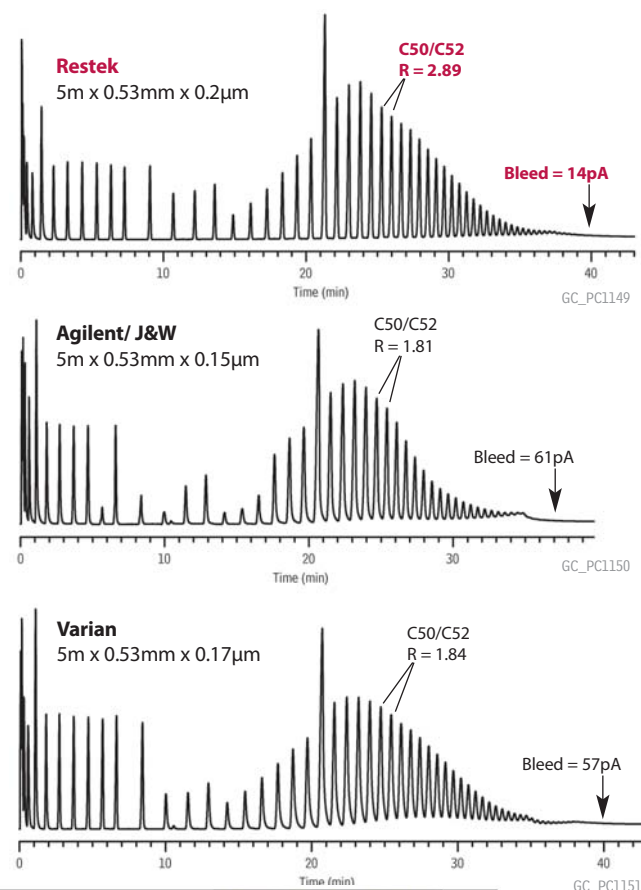


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**Figure 1** Low bleed, high efficiency MXT®-1HT SimDist columns outperform competitors (ASTM D6352 conditions).



### Lower bleed means:

- Longer column lifetime.
- More stable calibrations.
- Accurate boiling point determinations.

### Restek advantage:

Longer column lifetime and more accurate data!

### Higher efficiency means:

- Greater resolution; analyze more samples before method criteria are reached.
- Assured method performance.

### Restek advantage:

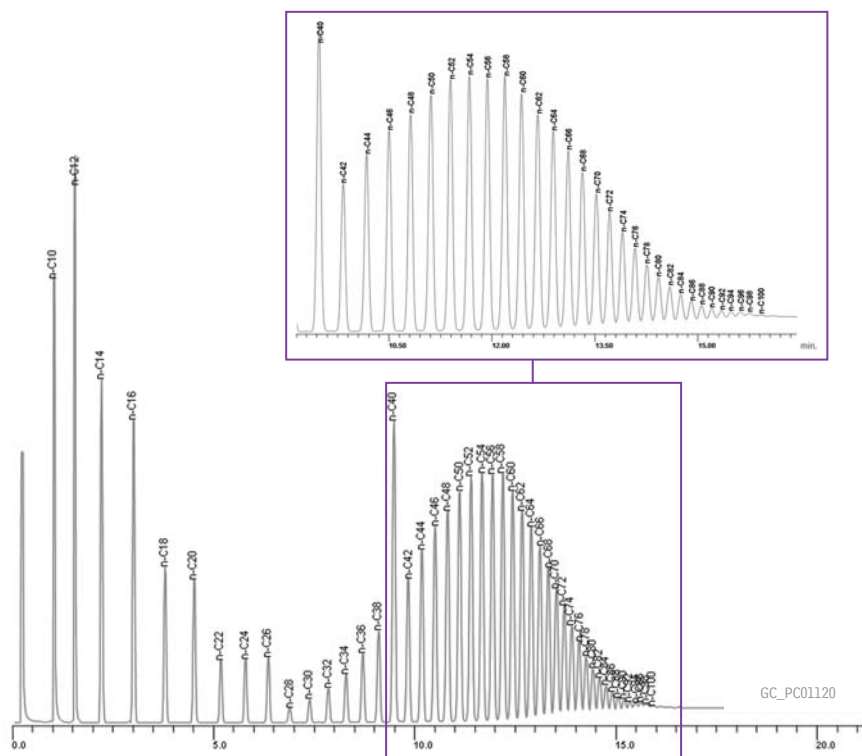
Run more samples within method specifications!

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**Figure 2** Robust MXT®-1HT SimDist columns meet all ASTM D6352 requirements, even under accelerated conditions.



**Column:** MXT®-1HT Sim Dist, 5m, 0.53mm ID, 0.20 $\mu$ m (cat.# 70115); **Sample:** C10-C100, 1% in carbon disulfide; **Inj.:** 0.2 $\mu$ L near on-column (PTV); **Inj. temp.:** 40°C to 430°C @ 100°C/min.; **Carrier gas:** helium, constant flow; **Flow rate:** 20mL/min.; **Oven temp.:** 40°C to 430°C @ 25°C/min.; **Det.:** FID @ 430°C; Chromatograms courtesy of Joaquin Lubkowitz, Separation Systems, Gulf Breeze, FL.

**Table I** Recommended SimDist columns (100% PDMS) for use in ASTM SimDist methods.

ASTM Method	Range	Recommended Column
D2887	C5-C44	5/10m x 0.53mm, df = 0.88 – 2.65 $\mu$ m
D7213 (2887-ext)	C5-C60	5m x 0.53mm, df = 0.15 – 1.2 $\mu$ m
D3710	Gasoline up to FBP 260°C (C14)	10m x 0.53mm, df = 2.65 $\mu$ m
D5307	Crude up to FBP 538°C (C42)	5m x 0.53mm, df = 0.2 $\mu$ m
D6352/D7500	C10-C90/C7-C110	5m x 0.53mm, df = 0.1 – 0.2 $\mu$ m
D7169	C5-C100	5m x 0.53mm, df = 0.2 $\mu$ m

FBP=final boiling point

**MXT®-1HT Sim Dist Column**  
(Siltek® treated stainless steel)  
(nonpolar phases)

ID	df ( $\mu$ m)	temp. limits	length*	cat. #
0.53mm	0.10	-60 to 430/450°C	5	70112
0.53mm	0.20	-60 to 430/450°C	5	70115
0.53mm	0.21	-60 to 430/450°C	10	70118
0.53mm	0.88	-60 to 400/430°C	5	70131
0.53mm	1.0	-60 to 380/400°C	10	70130
0.53mm	1.2	-60 to 380/400°C	10	70119
0.53mm	2.65	-60 to 360/400°C	10	70132
0.53mm	5.0	-60 to 360/400°C	10	70133

\*Length in meters



Al Carusone, Technical Service Specialist

## TECH TIP!

Oxygen and moisture will dramatically reduce siloxane phase stability, especially at temperatures over 400°C. To ensure maximum column lifetime, follow these guidelines for proper instrument set-up.



Use gas filters to remove oxygen and moisture from the carrier gas.

**See the triple filter special offer on page 8.**



When installing a column, prevent leaks by using a proper cutting device (such as a scoring wafer or MXT® tubing scorer) to ensure the column is not crushed. (cat. # 20523)



Use graphite ferrules for column installation; Vespel®/graphite ferrules may leak, due to expansion and contraction at high temperatures (>400°C).

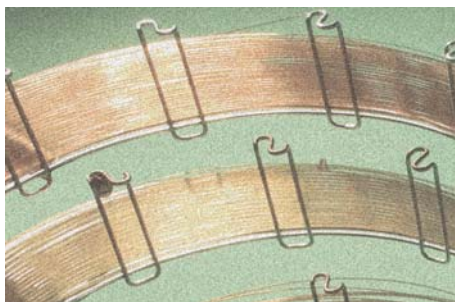
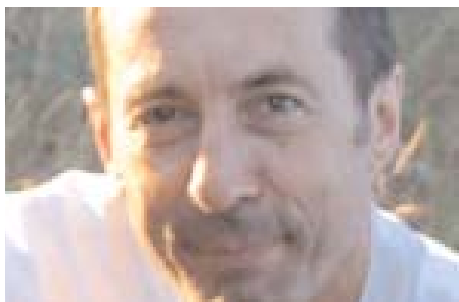


Check the system for leaks using an electronic leak detector. (cat. # 22839)



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## Unraveling Scent Signals to Protect African Wild Dogs

Peter Apps, Ph.D., Botswana Predator Conservation Trust

### Innovators in Chromatography

A continuing series of guest editorials contributed by collaborators and internationally recognized leaders in chromatography.

**Peter Apps** runs the BPCT Paul G. Allen Family Foundation Wildlife Chemistry Laboratory in Maun, northern Botswana. He is a zoologist with a long career in chromatography, a rare combination that led him back to his zoological roots to set up the laboratory in July 2008.

Although chromatography's versatility leads to its application to a host of diverse problems, helping to protect endangered African wild dogs from conflicts with people is perhaps not one that you would expect. With a grant from the Paul G. Allen Family Foundation, the Botswana Predator Conservation Trust (BPCT) has established a GC/MS laboratory to identify the chemical signals that African wild dogs use to mark their territory boundaries. The ultimate aim is to use artificial scent marks as "BioBoundaries" to limit movements by wild dogs into areas where they come into conflict with people and their livestock.

The BPCT BioBoundary project is led by Dr. John "Tico McNutt," who has been studying wild dogs since 1989, on the fringe of the Moremi Game Reserve and the Okavango Delta in northern Botswana. The GC/MS laboratory is located in the village of Maun, just 65 km from the BPCT study area, so that it can keep in close contact with field operations.

African wild dogs (*Lycaon pictus*) are intensely social predators. They live in packs of up to 27 adults and yearlings, in which usually only one pair breeds but everyone cares diligently for the pups. Numbering less than 6,000, they are one of Africa's most endangered carnivores, and their habitats are increasingly threatened by the expansion of human activities. Because wild dog packs have huge territories, only the very largest of protected wildlife areas can sustain viable populations. In Africa, wildlife areas with free-ranging carnivores are often separated from people and their livestock by only a line on a map or fences that are easily penetrated. Predators in livestock areas threaten peoples' livelihoods and the dogs' usual fate is to be shot, snared, or poisoned. The aim of the BPCT BioBoundaries project is to deploy artificial territorial scent marks, formulated with chemicals identified in natural wild dog marks, along protected area boundaries to create "virtual" neighboring packs that will deter dogs from crossing into areas where they are at risk. The stakes are high—population models predict that wild dogs will be extinct in the wild in 50 years, unless new ways are found to protect them.

Wild dogs, like nearly all mammals, live in a world dominated by odors. Airborne chemical signals, known as semiochemicals, play critical roles in their sexual and social behavior. The pack's dominant pair assiduously overmark each others feces and urine, and these double marks stake out the pack's territory.

Chemically, mammal scents are bafflingly complex, with the active messenger compounds at trace levels among hundreds of other components. Quantities of active compounds range down to picograms and concentrations of  $10^{-18}$  molar. Nonetheless, mammal chemical signals are within range of gas chromatography and mass spectrometry, as long as the technology is used to its full potential. Maximum resolution and reproducibility

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along with minimum contamination, discrimination, and limits of detection are required so that biological differences are not obscured by analytical artifacts and variability.

Sample preparation is both the most critical step and the Achilles heel. To preserve the integrity of the signal I have to sample what the dogs do: the volatiles in the air around a scent mark. Solid phase microextraction (SPME) and adsorption/thermal desorption looked promising, but yielded too many peaks from contaminants and too few from wild dogs. A simpler system was required to reduce contamination, variability, and analytical artifacts. Direct thermal desorption from urine-marked soil and cryotrapping with sample flow paths of glass and fused silica has provided the cleanest chromatograms so far. In nature, the scent marks are still active on hot, dry sand; therefore, samples can be dried prior to desorption to prevent icing of the cryotrap and then desorbed at 60°C.

The complexity of most mammal odors puts them well inside the Giddings zone, where at least 20% of chromatographic peaks overlap; not surprisingly, a dog mark chromatogram is so complex it has no clean baseline. Overlapping peaks cannot be properly quantified or identified and most failures to find an MS library match are due to coelutions that produce a mixed mass spectrum—only a minority of those without matches are new and, therefore, exciting compounds. To get cleanly resolved peaks, I will be using two-dimensional GC to transfer incompletely separated peaks from one column to another column with complementary selectivity.

Identifying everything in scent mark odor is unnecessary and impractical; the spotlight needs to fall on the few compounds that send the message. The critical challenge then is to differentiate the biologically relevant signal from the chemical noise, and this is where close links between the laboratory and the field operations play an absolutely critical role. Only dominant dogs produce territorial marks, so the signaling compounds will be present in their marks, but absent from subordinates' marks. The marks withstand 65K temperature differences in the soil substrate between midwinter midnights and summer afternoons. The marks last for at least six weeks and their emissions of territorial semiochemicals should be stable for at least as long. Without a detailed behavioral and social context for each sample it would be impossible to recognize the semiochemicals among the forest of extraneous peaks.

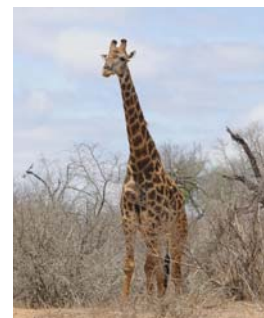
The wild dog boundary semiochemicals have to stand out against a background of the millions of natural chemicals that permeate the environment, and so I expect them not to be common constituents of mammal scent marks, feces or urine, or volatiles from plants or soil. Library searches of integer resolution mass spectra will eliminate compounds that are known to come from these sources.

Now that the sampling and separation conditions are worked out, in the months to come I will be running scent mark samples from several dogs in different packs searching for a peak, or a pattern of peaks that is present only in the marks of dominant animals, that stays the same with time and temperature, and that is not part of the environmental background. When I find it (or them) the next challenge will be to identify the compound(s). That will be a story for another time.

**For more information on the BPCT BioBoundaries project and African wild dog research, visit [www.bpctrust.org](http://www.bpctrust.org) or [www.wildentrust.org](http://www.wildentrust.org).**

## Travels in South Africa

Jack Cochran, Restek's Director of New Business and Technology, recently took these pictures on a photo safari while visiting South Africa to give seminars and collaborate on research projects. Jack was invited by ChromSA, the Chromatography Division of the South African Chemical Institute, to teach a course called, "Improving Your Gas Chromatographic Analyses." Following this and other speaking engagements at universities across the country, Jack spent several weeks working at the National Metrology Institute of South Africa on QuEChERS, GCxGC/TOFMS, PCB and dioxin analyses, on-column injection techniques, and various other gas chromatography projects at the invitation of Jayne de Vos.



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